

# PATENT SPECIFICATION

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## (54) HYDRAULIC FLUID COMPOSITION

(71) We, NIPPON OIL COMPANY LTD., a Japanese Body Corporate, of 3-12, 1-chome, Nishishinbashi, Minato-ku, Tokyo, Japan, and NISSAN MOTOR CO. LTD., a Japanese Body Corporate, of No. 2, Takaracho, Kanagawa-ku, Yokohama-shi, Kanagawa-ken, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a central system fluid composition which exhibits excellent physical and chemical properties such as high flash point, high boiling point, good fluidity at low temperatures, high shear stability and high viscosity index.

In order to meet the desire of the automotive industry to develop and use central hydraulic systems in vehicles, the Society of Automotive Engineers (SAE) of the U.S.A. have provided specifications for central system fluids, both petroleum base (SAE 71 R1) and synthetic base (SAE 71 R2). The main advantage of a central hydraulic system is that a single fluid, the central system fluid, is used to actuate several different hydraulically operated parts, thus making it possible to dispense with individual hydraulic systems for actuating, for example, brakes, power steering, wipers, air-conditioning, starting motors, clutches and hydropneumatic suspensions. Thus the size of the hydraulic systems to be accommodated in a vehicle can be reduced, and separate devices for cleaning exhaust gases or improving safety can be installed. Furthermore, still additional advantages such as the reduction of oil leakage troubles, easier maintenance, and utilisation of electronic circuits for the hydraulic system can be obtained.

The SAE 71 R1 specification combines many of the characteristics of power steering and brake fluids. The properties required in the specifications are as follows:

1. Good fluidity at low temperatures,
2. High shear stability,

- 3. Operability over a wide temperature range,
- 4. High boiling point and flash point,
- 5. Freedom from the formation of precipitates and/or condensates at low temperatures,
- 6. Little foaming,
- 7. High lubricating property and oxidation resistance, and
- 8. Freedom from causing corrosion of metal portions of the hydraulic system and excessive swelling or shrinkage of rubber parts.

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Because these requirements are rigorous, none of the presently commercially available central system fluids can yet pass all the tests set out in the SAE 71 R1 specification.

The present invention provides a central system fluid composition comprising 5 to 30% by weight of a viscosity index improver, and a hydrocarbon-type base oil, wherein at least 50% by weight of the hydrocarbon-type base oil is a chief base oil component selected from

- (A) polybutenes with an average molecular weight of 100 to 500;
- (B) homo- or copolymers with an average molecular weight of 100 to 500 of at least one  $\alpha$ -olefin containing 2 to 12 carbons, excepting butenes; and
- (C) hydrogenation products boiling at 250 to 380° C. of high aromatic components which are obtained by cracking petroleum oils,

and 0 to 50% by weight of the hydrocarbon-type base oil is a mineral oil; and wherein the viscosity index improver comprises at least one component selected from:

- (1) polymethacrylates with an average molecular weight of 50,000 to 200,000 and obtained by polymerising at least one ester of a saturated, monohydric aliphatic alcohol of 1 to 18 carbons with methacrylic acid; and
- (2) polymers with an average molecular weight of 10,000 to 200,000 obtained by

polymerising at least one olefin or diolefin of up to 5 carbons or aromatic vinyl compound.	nesium chloride, boron fluoride, or titanium tetrachloride, or complexes thereof, and if necessary also in the presence of a promotor such as an organic halide or hydrochloric acid.	70
5 The hydraulic fluid composition of the invention is the first and only product which fully satisfies the extremely rigorous SAE 71 R1 specification, and is particularly suited for use in a central hydraulic system of a vehicle. The composition of the invention is composed of the hydrocarbon-type base oil and the additives.	$\alpha$ -Olefin polymers which can be used as the chief base oil component are formed by homo- or co-polymerisation of at least one olefin of 2 to 12 carbons (excepting butene), particularly of 6 to 9 carbons. Of such $\alpha$ -olefin polymers those having a viscosity of not higher than 6.0 cSt at 100° F. and not higher than 2.0 cSt at 210° F. are suitably used. For this reason, the average molecular weight of the $\alpha$ -olefin polymers is 100 to 500, preferably 150 to 300. Those of average molecular weights less than 100 have objectionably low flash points. Those of average molecular weights greater than 500 show undesirably high viscosities. Any of the known methods for the preparation of $\alpha$ -olefin polymers may be employed, so far as the product meets the above requirements, for example, cationic polymerization in the presence of such catalysts as the aluminium chloride-aluminium bromide system, the aluminium bromide-hydrogen bromide system, the boron fluoride-alcohol system and the aluminium chloride-ester system; radical polymerization using heat or peroxide initiation or polymerization assisted by a Ziegler-type catalyst.	75
10 Hereinafter the composition of the invention will be more specifically explained.		80
15 1. Hydrocarbon-type base oil.		85
15 At least 50% by weight of the hydrocarbon-type base oil is a polybutene (A), $\alpha$ -olefin polymer (B) or hydrogenation product (C). The base oil preferably contains 80 to 100 weight % of (A), (B) or (C) or a mixture of at least two of these. The polybutenes (A) are the most preferred for use as the chief base oil component. When the base oil is a mixture of at least two of (A), (B) and (C), the viscosity of the mixture preferably is not higher than 6.0 cSt at 100° F. (37.8° C.), and not higher than 2.0 cSt at 210° F. (98.9° C.). If so desired, the base oil can contain up to 50% by weight of a mineral oil. Preferred mineral oils are those	90	
20	having a viscosity of not more than 6.0 cSt at 100° F. and not more than 2.0 cSt at 210° F., a viscosity index of not lower than 70, and a pour point of not higher than -30° C. The base oil may furthermore contain up to 50% by weight of a nuclear hydrogenation product of a heavy alkylbenzene.	95
25	The central systems fluid composition of the invention, in which the specified hydrocarbon-type base oil is employed, shows characteristically excellent fluidity at low temperatures.	100
30	Polybutenes which are to be employed as the chief base oil component according to the invention are commercially available polybutenes of average molecular weight ranging from 100 to 500, preferably 150 to 300. Those having an average molecular weight of less than 100 possess flash points which are too low while those having a molecular weights	105
35	greater than 500 have too high viscosities, and both fail to achieve the object of this invention. On the other hand, suitable viscosities are not higher than 6.0 cSt at 100° F., and not higher than 2.0 cSt at 210° F. and so far as the viscosity is kept within that range, minor amounts of higher molecular weight polybutenes or hydrogenated polybutene may be present in the compositions. The method of their preparation is well known to the experts. For example, a butane-butene fraction of the distillate collected in the procedure of naphtha cracking is used as a starting material, which is polymerised at -30° C. to 30° C. in the presence of a so-called Friedel-Crafts catalyst such as aluminium chloride, mag-	110
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45		120
50		125
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cSt at 100°F., and not higher than 2.0 cSt at 210°F., and a pour point not higher than -45°C. are preferably used.

2. Additives.  
 5 The central system fluid composition of the invention is prepared by mixing the above-described hydrocarbon-type base oil with a specific viscosity index improver as the additive, and if necessary also with such other additives as an antioxidant, detergent, metal deactivator, antifoaming agent or rubber swelling agent. We found that in order to achieve the object of this invention to provide the central system fluid satisfying the SAE 71 R1 specification, the selection of an adequate viscosity index improver is most important. Specifically, the viscosity index improver to be employed in this invention should possess high shear stability, high viscosity-increasing effect but at such low temperature as -40°C., low viscosity-increasing effect. The viscosity index improver used in accordance with the invention is at least one of:

25 (1) polymethacrylates with an average molecular weight ranging from 50,000 to 200,000, which are obtained by polymerizing at least one ester of a saturated, monohydric straight-chain or branched-chain aliphatic alcohol of 1 to 18 carbons with methacrylic acid,

30 (2) polymers with an average molecular weight ranging from 10,000 to 200,000, preferably 40,000 to 100,000, which are obtained by polymerizing at least one olefin or diolefin of up to 5 carbons, or aromatic vinyl compound such as styrene (for example, ethylene-propylene copolymer, isobutylene homopolymer, butadiene-styrene copolymer and isoprene-styrene copolymer).

40 While it is permissible to use mixtures of the above components (1) and (2) in optional blend ratios as the viscosity index improver according to the present invention, the component (1) is the more preferred. The viscosity index improver in this invention is used in a proportion of 5 to 30 wt.%, preferably 10 to 20 wt.%, based on the total weight of the composition.

45 According to the invention, various additives used in the art, other than the viscosity index improver, may be used if necessary. The weight percents indicated for each type of additives hereinbelow are all based on the total weight of the composition.

50 As the antioxidant, for example, metal salts of dialkyldithiophosphoric acids, phenyl- $\alpha$ -naphthylamine, 2,6-di-t-butyl-p-cresol and 2,6-di-t-butylphenol are preferred, which may be used singly or as mixtures. The suitable amount of the antioxidant ranges from 0.1 to 3.0 wt.%.

55 As the detergent, for example metal-containing compounds such as neutral metal sulfonates, basic metal sulfonates, superbasic

metal sulfonates, metal phenates and metal phosphonates and ashless dispersants such as alketyl succinimides and benzylamines may be named. They can be used singly or as mixtures in an amount of 0 to 4.0 wt.%.

60 Besides the foregoing, a metal deactivator such as benzotriazole may be added in an amount within the range of 0.005 to 0.5 wt.%, and an antifoaming agent such as silicone or ester-type antifoaming agents, for example, a low molecular weight polymethacrylate, may be added in an amount of up to 0.5%. Furthermore, when a polybutene or  $\alpha$ -olefin polymer is used as the base oil, nitrile rubber undergoes shrinkage. In order to prevent it, an aromatic compound as a rubber-swelling agent may be added, if necessary, in an amount of 1 to 3 wt.%. The total sum of the above various additives excepting the viscosity index improver should be 0.1 to 10 wt.%, and within said range optional ratios can be selected so far as no deleterious effect is produced on the viscosity characteristics and fluidity at low temperatures.

65 As already mentioned, the composition of the present invention is the first and only hydraulic fluid composition satisfying all the requirements of the SAE 71 R1 specification. The composition is particularly suited for use as a central system fluid, but also can have other uses such as brake fluid, shock-absorbing fluid and automatic transmission fluid.

70 Hereinafter the invention will be more specifically illustrated with reference to working examples.

#### Example 1.

Composition	Percent by Weight
Polybutene A note 1)	78.0
note 2)	105
Viscosity index improver	18.4
Antioxidant (zinc di-2-ethylhexyldithiophosphate)	0.9
Detergent (calcium sulfonate and calcium phenate)	2.1
Metal deactivator (2,5-di-mercapto-1,3,4-thiadiazole)	0.5
Antifoaming agent (low molecular weight polymethacrylate)	0.1

Note 1) "Polybutene A" has an average molecular weight of approximately 250, and a viscosity of 4.88 cSt (100°F.) and 1.59 cSt (210°F.).

Note 2) The polymethacrylate of average molecular weight of 143,000, which is obtained by polymerizing an ester of saturated monohydric aliphatic alcohol of 1—18 carbons con-

taining not less than 60 wt.% of n-dodecyl alcohol with methacrylic acid.

Example 2.

	Composition	Percent by Weight
5	Polybutene A note 3)	75.8
10	Polybutene B note 4)	4.0
15	Viscosity index improver Antioxidant (same as that employed in Example 1) Detergent (same as that employed in Example 1) Metal deactivator (same as that employed in Example 1) Antifoaming agent (same as that employed in Example 1)	16.6 0.85 2.2 0.5 0.05

Note 7) A paraffinic hydrocarbon oil which has been dewaxed at low temperature -40°C.  
Note 8) The same polymethacrylate as employed in Example 1.

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Example 4.

	Composition	Percent by Weight
5	$\alpha$ -Olefin polymer note 9)	80.8
10	Viscosity index improver Antioxidant (the same as that used in Example 3) Detergent (the same as that used in Example 3) Metal deactivator (the same as that used in Example 3) Antifoaming agent (the same as that used in Example 3)	16.0 0.7 2.2 0.28 0.02
15		65
20		70

20 Note 3) The details are the same as those of Example 1.

25 Note 4) "Polybutene B" has an average molecular weight of approximately 310, and a viscosity 21 cSt (100° F.) and 4.0 cSt (210° F.).

The mixture of polybutene A and polybutene B has a viscosity of 5.21 cSt (100° F.).

30 Note 5) A mixture of 20 parts of the polymethacrylate employed in Example 1 and 80 parts of an ethylene/propylene copolymer of average molecular weight of 50,000.

	Example 3.	Percent by Weight
35	Composition	
	Polybutene A note 6)	53.7
40	Mineral oil note 7)	28.9
45	Viscosity index improver Antioxidant (2,6 - di - t - butyl p - cresol) Detergent (alkyl succinimide) Metal deactivator (2,5 - di - mercaptor - 1,3,4 - thiadiazole) Antifoaming agent (low molecular weight polymethacrylate)	14.1 0.95 2.2 0.1 0.05

Note 9) The b.p. 65—250° C./0.4 mmHg fraction of the distillate from the product of octene-1 polymerization at atmospheric pressure and 30° C., in the presence of a normal Ziegler-type catalyst, having a molecular weight of 320 and the viscosity of 1.95 cSt (210° F.).

Note 10) The same polymethacrylate as used in Example 1.

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Example 5.

	Composition	Percent by Weight
5	$\alpha$ -Olefin polymer note 11)	80
10	Hydrogenation product of high aromatic components note 12)	85
15	Viscosity index improver	15.0
20	Antioxidant (the same as that used in Example 1)	0.7
25	Detergent (the same as that used in Example 1)	2.2
30	Metal deactivator (the same as that used in Example 1)	0.1
35	Antifoaming agent (silicone-type)	20 ppm
40		95

50 Note 6) The details are the same as those of Example 1.

Note 11) The b.p. 210—370° C. fraction of distillate from the heavy oil obtained by thermal cracking of naphtha, is hydrorefined and further hydrogenated. By distillation, a fraction boiling at 265—307° C. is collected. The reaction conditions and the properties of the hydrogenation product are as follows:

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	Hydrorefining	Hydrogenation
Catalyst	nickel-molybdenum-alumina	nickel-diatomaceous earth promoted by chromium and copper (nickel content, 45%)
Reaction Pressure (Kg/cm <sup>2</sup> )	35	70
Reaction Temp. (°C.)	330	200
Hydrogen/Oil molar ratio	3.5	16
LHSV	3	1.0

Specific gravity d <sub>4</sub> <sup>20</sup>	0.9349
Refractive index n <sub>D</sub> <sup>20</sup>	1.5056
Average molecular weight	200
Viscosity cSt (100°F.)	5.150

Note 12) The same polymethacrylate as used in Example 1.

5 Table 1 shows the results obtained by subjecting the composition of Example 1 to the standard tests of central system fluid based on the SAE 71 R1 specification. The composition passed all the tests.

Similarly, the compositions of Examples 2 to 5 were tested and passed all the items with satisfactory results.

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TABLE I

No.	Test Items	Test Method	SAE 71 R1 Specification	Test Results	J Judgment
1	Viscosity-Kinematic	Based on the SAE 71 R1 specification.	200 cSt max. at -40°F. (before and after shear) as determined by Low Temperature (-40°F.) Viscosity-Brookfield Procedure	1860 cSt  5.5 cSt min. at 210°F (after shear) as determined by the procedure outlined by ASTM D 445.	passed
2	Flash Point	do	225°F. (107.2°C.) min. as determined by the ASTM D 92 method	118°C.	passed
3	Initial Boiling Point	do	400°F. (204°C.) min. as determined by the ASTM D 158 method	226°C	passed
4	Cold Test	do	The sample should be transparent and show no stratification or sedimentation after 6 days' standing at -50°F. in accordance with SAE J 70R3 method	Satisfactory	passed

TABLE 1 (cont.)

No	Test Items	Test Method	SAE 71 R1 Specification	Test Results	Judgment
5	Fluidity and Appearance at Low Temperature (Brake Fluid)	after 6 days standing at -40°C., the air bubble shall rise to the top of the fluid in not more than 10 sec. upon inversion of the sample bottle according to SAE 70R3 method  After 6 hours' standing at -50°C., the air bubbles shall have a rise velocity of no more than 35 sec.	—  1 sec.	5.4 sec.	passed  passed
6	Foaming	Based on SAE 71 R1 specification	i) 100 ml foam volume max. at end of 5-minute blowing period  ii) No foam left at end of 4-minute settling period; as determined by ASTM D 892 method	85 ml  No foam after the settling	passed

TABLE 1 (cont.)

No.	Test Items	Test Method	SAE 71 R1 Specification	Test Results	Judgment
7	Corrosion Resistance	Based on SAE 71 R1 specification	As determined by ASTM D 665 turbine oil test with distilled water	no rust	passed
8	Seal compatibility (Rubber Swelling)	Based on SAE 71 R1 specification	Based on SAE 70 R3, the increase in the base diameter of nitrile rubber cups after 70 hours' immersion in sample liquid at $250 \pm 5^{\circ}\text{F}$ . shall not be less than 0.125 mm, nor more than 1.375 mm	0.755–1.371 mm	passed

The rubber surface shall not be tacky or show any sloughing as may be indicated by carbon black on the surface.

Satisfactory

TABLE 1 (cont.)

No.	Test Items	Test Method	SAE 71 R1 Specification	Test Results	Judgment
9	Shear Test*				
	(a) Power steering Pump Test	5.5 cSt min. after 100 hours operation of pump. at 65.5°C. pump entrance temperature. See SAE Recommended Practice, SAE J72		6.52 cSt (210°F.)	passed
	(b) High-pressure Vane Pump Test (of hydraulic oils)	Based on ASTM D-2882-70T	Operation time after breaking in (hrs.)	Viscosity (cSt, 98.9° C.)	passed
			0	7.87	
			20	7.58	
			66	7.30	
			100	7.02	
	(c) Sonic Shear Stability (of polymer-containing oils)	Based on ASTM D-2603	-	Viscosity at end of 4 hours' irradiation 6.15 cSt (210°F.)	passed
				Viscosity reduction ratio 20.7%	

TABLE 1 (cont.)

No.	Test Items	Test Method	SAE 71 R1 Specification	Test Results	Judgment
10	Anti-wear	Based on SAE 71 R1 Specification	Pump delivery at 700 rpm and 600 psi discharge shall not decrease more than 0.2 gpm during 100 hrs. as indicated by measurements on Standard Reference Fluid at start and end of test as determined by Wear and Pump Delivery Test  Pump parts, by visual inspection, shall show no signs of excessive wear. Parts should be burnished and show no signs of galling.	Pump discharge normal, no wear observed	passed
11	Shell high speed fourball test 1800 rpm 1 hr.	—	—	The sample had equivalent wear characteristics with commercial hydraulic oil as below	passed

Load (kg)	Central system fluid of Example 1	Commercial hydraulic fluid	
		A	B
30	0.45	0.40	0.36
40	0.55	0.69	0.45
50	0.83	0.72	0.76
		Unit; wear marks mm	

TABLE 1 (cont.)

No.	Test Items	Test Method	SAE 71 RI Specification	Test Results				Judgment
				1. Property Change of Sample Oil				
				Time (hr)	0	100	200	300
12	Oxidation Stability	Total rating 80 min. evaluation of clean- ness at end of 300 hrs. measurements at 275°F. using an automatic trans- mission, based on SAE J72.	—	Viscosity cSt (100°F)	25.40	25.21	25.54	25.54
				Viscosity cSt (210°F)	7.74	7.57	7.57	7.43
				Acid value (mg KOH/g)	1.42	1.16	1.29	1.48
				Base value (do.)	2.66	1.13	0.98	0.77
				Insolubles in petroleum ether (wt. %)	0.01	0.14	0.17	
				Insolubles in benzene (wt. %)	0	0.08	0.11	

TABLE 1 (cont.)

No.	Test Item	Test Method	SAE 71 R1 Specification	Test Results		Judgment
				2. Cleanness Evaluation (10 at best)		
				Varnish *1	Sludge *2	
	Turbine outer surface			10	-	
	Converter housing (outer)			-	10	
	Screen			10	10	passed
	Steel clutch plates			10	-	
	Valve body (Outer)			10	-	
	Valve body (cavities)			-	10	
	Clutch piston			-	10	
	Clutch cylinder			-	9.9	
	Bottom of transmission			10	-	
	Total			50	49.9	

\*1. Based on CRC Manual No. 9.

\*2. Based on CRC Manual No. 10.

3. As above-indicated, substantially no viscosity change was observed with time passage. When the infrared absorption spectrum of the fluid before use is compared with that of the fluid at the end of 300 hours' use no infrared absorption is observed due to oxidation product formed by the deterioration of the oil. The cleanliness evaluation was as high as 99.9.

\* Concerning the ninth test item, "Shear Test", the test apparatus designated by the SAE 71R1 specification was unavailable. Accordingly, the judgments were made by substituting the equivalent test methods (a), (b) and (c) for the specified methods.

**WHAT WE CLAIM IS:—**

1. A central system fluid composition comprising 5 to 30% by weight of a viscosity index improver and a hydrocarbon-type base oil, wherein at least 50% by weight of the hydrocarbon-type base oil is a chief base oil component selected from

- 5      (A) polybutenes with an average molecular weight of 100 to 500;
- 10     (B) homo- or copolymers with an average molecular weight of 100 to 500 of at least one  $\alpha$ -olefine containing 2 to 12 carbons, excepting butenes; and
- 15     (C) hydrogenation products boiling at 250 to 380° C. of high aromatic components which are obtained by cracking petroleum oils;

20     and 0 to 50% by weight of the hydrocarbon-type base oil is a mineral oil; and wherein the viscosity index improver comprises at least one component selected from:

- 25     (1) polymethacrylates with an average molecular weight of 50,000 to 200,000 and obtained by polymerising at least one ester of a saturated, monohydric aliphatic alcohol of 1 to 18 carbons with methacrylic acid; and
- 30     (2) polymers with an average molecular weight of 10,000 to 200,000 obtained by polymerising at least one olefin or diolefin of up to 5 carbons or aromatic vinyl compound.

2. A composition according to claim 1 wherein the chief base oil component provides 80 to 100% by weight of the hydrocarbon-type base oil.

- 3. A composition according to claim 1 or 2 wherein the chief base oil component has a viscosity of not more than 6.0 cSt at 100° F. and not more than 2.0 cSt at 210° F.     35
- 4. A composition according to claim 4 wherein the chief base oil component is a polybutene having an average molecular weight of 150 to 300.     40
- 5. A composition according to any one of the preceding claims wherein the mineral oil when present has a viscosity of not more than 6.0 cSt at 100° F. and not more than 2.0 cSt at 210° F., a viscosity index of at least 70, and a pour point of not higher than -30° C.     45
- 6. A composition according to any one of the preceding claims wherein the viscosity index improver content, based on the total weight of the composition, is from 10 to 20% by weight.     50
- 7. A composition according to any one of the preceding claims which also contains up to 10% by weight of at least one additive selected from antioxidants, detergents, metal deactivators, antifoaming agents and rubber swelling agents.     55
- 8. A composition according to claim 1 substantially as described in any one of the Examples.     60

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